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Real Time Study of Cement Hydration

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Beamline(s): X7B

Introduction: Alumina cements and Portland cements are multicomponent systems where phase composition and qualitative analyses can be studied by in situ synchrotron X-ray powder diffraction analysis. This technique can also be applied to real time studies of cement hydration processes, where the rate of depletion and formation of phases can be established. The rate of reaction of the cement component $Ca_3Al_2O_6$ with water has been studied by neutron powder diffraction [1] and by energy dispersive synchrotron X-ray powder diffraction [2]. The aim of the present investigation was to study the hydration of a real industrially produced cement using constant wave length synchrotron X-ray powder diffraction.

Methods and Materials: The sample of cement used had a high content of cubic $Ca_3Al_2O_6$, and its original content of calcium silicates was extracted with a salicyclicacid-methanol solution. The real time investigations of the hydrolysis reactions were made using the MAR-diffractometer at beam line X7B, NSLS. The wave length was λ = 0.90731 Å, and the samples were kept in 0.7 mm diameter quartz glass capillaries. The solid cement was placed in the tip of the capillary, and water was placed in the capillary without contact with the solid sample. After a few diffraction patterns had been recorded, the capillary was pressurized from a nitrogen gas cylinder to an internal pressure of 1700 kPa, and by this pressure the water was pressed into contact with the solid.

Results: The figure displays powder diffraction patterns of the reaction mixture heated in a temperature ramp from 25 to 75°C. The solid was mixed with water after the second scan was recorded. The depletion of $Ca_3Al_2O_6$ starts immediately, and formation of two phases can be observed. However, they are not stable at elevated temperatures. The phase C_4AH_{19} has its maximum at 40°C, and the phase C_2AH_{10} at 48°C. The final product C_3AH_6 starts to be formed when the C_4AH_{19} phase intensities start depletion, that is C_3AH_6 grows in consumption of C_4AH_{19}. The results obtained in this investigation are different from the hydrolysis reactions of C_3A previously reported.[2] The cement chemical nomenclature used above gives the composition of compounds as a combination of oxides; C_3A = $3CaO{\cdot}Al_2O_3$, C_2AH_{10} = $2CaO{\cdot}Al_2O_3{\cdot}10H_2O$, C_3AH_6 = $3CaO{\cdot}Al_2O_3{\cdot}6H_2O$, and C_4AH_{19} = $4CaO{\cdot}Al_2O_3{\cdot}19H_2O$.

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References: [1] A. N. Christensen and M. S. Lehmann, *J. Solid State Chem.*, **51**, 196 (1984).
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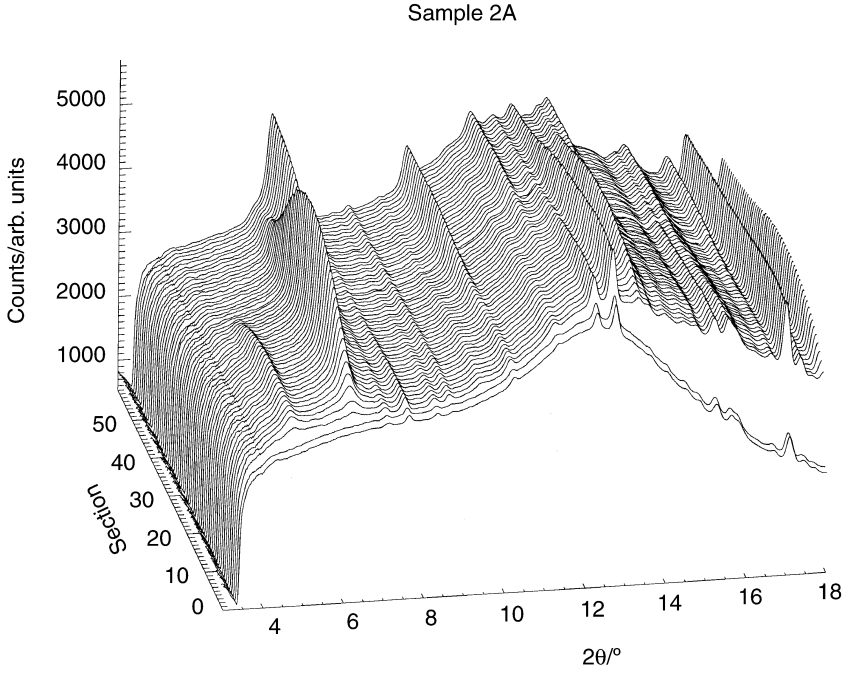

Figure 1: Stack of powder diffraction patterns of the reaction mixture heated in a temperature ramp from 25 to 75°C. The solid was mixed with water after the second scan which significantly chances the background.